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Nonpolar a -plane ZnO films grown on GaN/sapphire templates with SiN_x interlayer by pulsed laser deposition

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Abstract

Nonpolar a -plane ZnO films were deposited for the first time on nearly lattice-matched a -plane GaN templates using an *in situ* SiN_x interlayer by the pulsed laser deposition method. The symmetric and asymmetric reciprocal space mappings reveal the broadening effects of the reciprocal lattice points and the residual biaxial in-plane tensile strain of a -plane ZnO to be 0.335% and 0.055% along the $[0001]$ c -axis and $[1\bar{1}00]$ p -axis, respectively. The photoluminescence spectrum at 85 K is dominated by neutral donor-bound excitons and free-electron-to-bound ($e\text{-}A_0$) emissions; relatively intense LO-phonon replicas of ($e\text{-}A_0$) have also been observed in a -plane ZnO. Temperature-dependent PL spectra have also been discussed to identify the origin of the emission peaks. Up to fourth $A_1(\text{LO})$ phonon mode can be observed to be enhanced significantly from the resonant Raman spectrum.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Among the available wide band gap materials, zinc oxide (ZnO) is a very attractive material for a large variety of promising applications due to its wide direct band gap of 3.37 eV, large free exciton binding energy (60 meV), high mechanical and thermal stabilities. The difficulty of p-type doping in ZnO has impeded the fabrication of ZnO p-n homojunction devices. Besides, due to the absence of cheap, large area, native substrates, fabrication of ZnO has been carried out mainly on highly mismatched substrates such as sapphire. Lattice mismatch between an epilayer and a substrate plays a crucial role in heteroepitaxy [1]. To improve

the crystalline quality, it is highly desirable to choose a suitable substrate that exhibits a smaller lattice mismatch with ZnO. For technological applications, it is highly desirable to grow ZnO on a conductive or a semi-conductive substrate in order to fabricate heterostructure devices and to make electronic measurements. Also, we note that ZnO/GaN heterojunction has been suggested as a strong candidate for device applications, since both have wurtzite crystal structures with small lattice mismatches (0.4% for the c -axis and 1.9% for the a -axis) and similar thermal expansion [2]. To utilize the excellent optical and electronic properties of ZnO/GaN devices, one issue common to both ZnO and GaN is the need to grow lower defective epitaxial layers. In general, ZnO films have been grown on GaN templates using different

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methods including molecular beam epitaxy (MBE), metal-organic chemical vapour deposition (MOCVD), sputtering, pulsed laser deposition (PLD) and so on [3–5]. However, most of these studies dealt with conventional *c*-plane oriented thin films with strong piezoelectric and spontaneous polarizations. Therefore, in order to control the polarization effect, it is preferable to grow nonpolar ZnO surfaces to eliminate the quantum confined Stark effect, such as *a*-plane (11 $\bar{2}$ 0) and *m*-plane (1 $\bar{1}$ 00). The use of GaN with a nonpolar plane could be effective in solving these problems. Compared with the isotropic in-plane lattice mismatch observed in *c*-plane ZnO, anisotropic in-plane lattice mismatch and thermal mismatch of ZnO and GaN result in nonpolar ZnO surfaces with in-plane structural, optical, acoustic and electrical anisotropic properties, thus playing a key role in novel designs of both electronic and optoelectronic devices, such as surface acoustic wave devices and ultraviolet light modulators [6].

It is already well known that growing high quality, pit-free nonpolar films is very difficult compared with polar films. Only recently, several research groups have reported the growth of *a*-plane ZnO on *r*-plane sapphire, (001) LaAlO₃, but the production of high quality material has not been achieved so far [7–9]. In fact, Hong *et al* have discussed the growth characteristic and properties of nonpolar GaN and ZnO films. Due to the high lattice mismatch (with ZnO $\sim$ 18.3% and GaN $\sim$ 16.1% along [1 $\bar{1}$ 00]) the most commonly used substrate, *r*-plane sapphire, could significantly reduce the nonpolar ZnO and GaN crystalline quality with a high density of defects. The difficulty in growing these nonpolar films comes from the planar anisotropic nature of the growing surface. The anisotropic in-plane strain and the adatom diffusion length considerably modify the growth front, resulting in a wavy, stripe-like growth feature [10]. Moreover, Kobayashi *et al* demonstrated that single crystalline *a*-plane GaN with an atomically flat surface could be grown on *a*-plane ZnO substrates by PLD [11]. Accordingly, it is very interesting and necessary to study the growth of nonpolar ZnO materials on nonpolar or semipolar GaN. Up to now, there has been little work related to nonpolar ZnO grown on nonpolar GaN templates. In addition, hardly any work on the full strain state of nonpolar ZnO by reciprocal space mappings (RSMs) and the resonant Raman scattering (RRS) optical property has been reported. In this paper, we mainly present the epitaxial growth of high quality nonpolar *a*-plane ZnO on nearly lattice-matched *a*-plane GaN templates with an *in situ* SiN_x nanomask for defect reduction. The variation in the structural and optical properties of *a*-plane ZnO is discussed in detail. We think that this study will be helpful in developing a deep understanding of the nonpolar ZnO material growth and potentially related electronic and optoelectronic devices' design.

2. Experimental procedure

The investigated ZnO thin films were prepared on nonpolar *a*-GaN/*r*-Al₂O₃ templates using a PLD system. For our study, firstly, *a*-plane GaN layers with and without SiN_x were grown on *r*-plane sapphire substrates in a Thomas Swan MOCVD system. The *in situ* SiN_x layer was inserted after

a low temperature nucleation layer by flowing disilane and ammonia. Epitaxial GaN templates of approximately 1.8 μ m thickness were grown at about 1070 °C using trimethylgallium and ammonia as precursors. Hydrogen was used as the carrier gas. The reactor pressure was maintained at about 200 Torr. Subsequently, as-grown GaN/sapphire templates were used to deposit the thin ZnO layer in a PLD system. The laser beam (an excimer KrF laser source, with a pulse duration of 20 ns and a frequency of 5 Hz) operated at an energy density of 1.5 J cm^{−2} was focused through a 60 cm focal lens onto a rotating sintered bulk ZnO target. The substrate temperature was held at about 600 °C during growth. The typical thickness of the resultant ZnO films was determined to be around 700 nm by the surface profiler.

The crystallinity and orientation of the as-grown samples were investigated using high resolution x-ray diffraction (XRD) methods (Panalytical X'pert Pro MRD) for both rocking curves (RCs) and RSMs measurements. The photoluminescence (PL) spectra were performed in the temperature range 85–290 K to evaluate the optical properties of the sample by using the 325 nm line of a He–Cd laser as the excitation source. The RRS spectrum was measured in backscattering geometry by a Jobin Yvon-HR800 spectrometer using a He–Cd laser.

3. Results and discussion

In this study, we found that the best crystalline quality of ZnO film on a GaN template was obtained at a substrate temperature around 600 °C by PLD. The growth of ZnO at a higher temperature around 700 °C resulted in the formation of polycrystalline materials, probably due to the interfacial reaction between ZnO and GaN or the re-evaporation of the adatoms from the film surface at a high substrate temperature [12]. Further details of the growth procedure will be described elsewhere. Typical XRD 2 θ / ω scan patterns of the ZnO films deposited on *a*-GaN/*r*-sapphire templates with and without the SiN_x interlayer at a growth temperature of 600 °C by PLD are shown in figure 1. Except for the narrow {1 $\bar{1}$ 02} peaks from the *r*-plane sapphire substrate, only the peaks of nonpolar *a*-plane (11 $\bar{2}$ 0) GaN and *a*-plane (11 $\bar{2}$ 0) ZnO were observed in the 2 θ full range from 20° to 80°, establishing an out-of-plane growth orientation relationship of (11 $\bar{2}$ 0) ZnO $\parallel$ (11 $\bar{2}$ 0) GaN $\parallel$ (10 $\bar{1}$ 2) sapphire. This demonstrates that unique *a*-plane preferred orientation ZnO films can be obtained on *a*-plane GaN template layers at 600 °C by PLD. Also, using the GaN template with an SiN_x interlayer (figure 1(b)) has proved effective in improving the crystal quality of *a*-plane ZnO. Omega x-ray RCs were measured for *a*-plane ZnO and GaN films, both on-axis (11 $\bar{2}$ 0) and off-axis (10 $\bar{1}$ 0) reflections. Here, the azimuth angle 0° is defined with the x-ray incident geometry parallel to the *c* [0001] direction and the sample is rotated clockwise. Compared with the RC full width at half maximum (FWHM) values of 0.71° and 0.74° for the ZnO film on GaN template without SiN_x, the FWHMs on-axis (11 $\bar{2}$ 0) at azimuth 0° and 90° (*c*- and *m*-mosaics, respectively) for ZnO grown on a GaN template with SiN_x were about 0.40° and 0.56°, suggesting that the crystalline quality

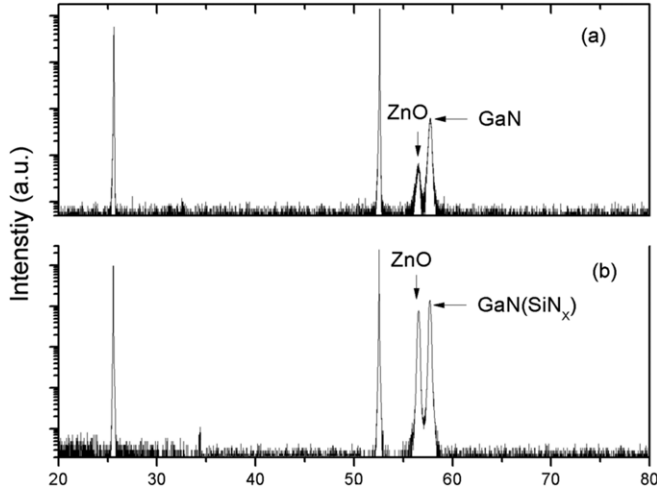


Figure 1. XRD $2\theta/\omega$ scan patterns of ZnO films fabricated by PLD at a growth temperature of 600 °C on GaN/sapphire templates without SiN_x interlayer (a) and with SiN_x interlayer (b).

of *a*-plane ZnO is comparable to that directly on *r*-sapphire by plasma-assisted MBE [7]. Meanwhile, the FWHMs for the GaN template with the SiN_x interlayer were 0.28° and 0.42° at azimuth 0° and 90°, respectively. The M shape of the RC values as a function of azimuth angles indicates the low *C*_{2v} symmetry in the growth *a*-plane. The (10 $\bar{1}$ 0) off-axis peak, which measures the ‘twist’ mosaic, had FWHMs of 0.58° for ZnO and 0.48° for GaN. These small FWHM values are in agreement with a much lower dislocation density, similar to a previous observation in planar *a*-plane GaN [13]. From the analysis above, we conclude that better crystalline quality *a*-plane ZnO can be fabricated by PLD on a GaN template with an SiN_x interlayer. These data indicate that the ratio of *c*-mosaic to *m*-mosaic approached unity for (11 $\bar{2}$ 0) RC ZnO on GaN template with and without the SiN_x interlayer, mainly due to the smaller mismatch between ZnO and GaN. The following discussion concentrates on higher quality ZnO films grown on GaN templates with an SiN_x interlayer. The RC broadening feature of *a*-plane ZnO is also approved from the symmetric (11 $\bar{2}$ 0) RSMs at two positions, azimuth 0° and 90°, as shown in figures 2(a) and (b). The upper ellipse is the RSM of (11 $\bar{2}$ 0)_{GaN} and the lower one is the RSM of (11 $\bar{2}$ 0)_{ZnO}. The FWHM values of ZnO in reciprocal space parallel to the *Q_x*-axis in RLU are 29.00×10^{-4} for azimuth 0° and 39.97×10^{-4} for azimuth 90°, while along the *Q_y*-axis they are 8.56×10^{-4} and 8.71×10^{-4} , respectively. This clearly indicates that several factors contribute to the RC width with different weights, depending on the sample orientation relative to the scattering plane. Typically, in the case of the symmetric geometry, it is assumed that the reciprocal lattice point significantly broadened in the lateral direction likely due to the contribution by the anisotropic defect distribution, bending, mosaic tilt and small lateral coherence lengths of the mosaic blocks [14].

To get more information regarding interplanar spacings and to determine the full state of strain in the *a*-plane ZnO grown on the GaN template, we define the wurtzite crystal on a new set of reference axes, with periodicity *a*[11 $\bar{2}$ 0]

(along the *a*-axis), *p*[11 $\bar{1}$ 00] (along the *m*-axis) and *c*[0001] (along the *c*-axis), respectively, justified from a fundamental point of view by the surface atomic structure of *a*-plane ZnO. This axis transformation is equivalent to describing the lattice deformation in an orthorhombic system rather than in the initial hexagonal one. Figures 2(c) and (d) are asymmetric reciprocal lattice space mappings (11 $\bar{2}$ 2) and (20 $\bar{2}$ 0). These reciprocal lattice points of ZnO are elliptical with their major axes aligned along or close to the ω scan directions. The broadening effects of lateral correlation length and tilt are predominantly caused by the limited mosaic block dimensions and an additional mosaic tilt. Interestingly, it is found that ZnO is partially relaxed in the *p*-axis direction, while the lattice is almost coherent in the *c*-axis direction, quite similar to a previous paper on *a*-plane GaN grown on ZnO substrates [11]. By calculating from the symmetric and asymmetric RSMs of selected (11 $\bar{2}$ 0), (11 $\bar{2}$ 2) and (20 $\bar{2}$ 0) reflections, the interplanar spacing parameters are obtained independently along each orthogonal direction [15, 16]. Thus, the in-plane and out-of-plane strain components can be deduced by

$$\varepsilon = (d_{\text{measure}} - d_{\text{relax}})/d_{\text{relax}}.$$

The results of calculations of the above equation show that the *a*-plane ZnO film possesses an in-plane tensile strain of +0.335% and +0.055% along the *c*[0001] and *p*[11 $\bar{1}$ 00] directions, respectively. This indicates that only 10.9% of the strain in the *c*[0001] direction is released, while 96.6% of that in the *p*[11 $\bar{1}$ 00] direction is released, which implies that the energy barriers for the introduction of misfit dislocations are different for perpendicular crystallographic directions. Thus, it is reasonable to consider that *a*-plane ZnO heteroepitaxial layers are subject to residual strain induced by the lattice mismatch between the substrate and the deposited layer as well as the difference in the thermal expansion coefficients of the substrate and the layer material. This has to be taken into account in order to develop high quality devices, because strain and strain relaxation significantly affect the properties of semiconductor structures. Further studies are ongoing through advanced growth techniques such as SiO₂-masked epitaxial lateral overgrowth (ELOG) and ZnO lower temperature layer to improve the *a*-plane ZnO film crystalline quality and surface morphology.

We now discuss the PL properties of the *a*-plane ZnO thin film at various temperatures. The representative PL spectrum of the nonpolar ZnO film at 85 K is shown in figure 3. Luminescence extending from the band edge to the yellow spectral range can be detected by strong higher energy emission lines in the vicinity of 3.3 eV and a relatively weak lower energy broadband centred at 2.3 eV, implying good optical quality of the *a*-plane ZnO film. It is well known that in ZnO films the UV emission band could be related to a near band-edge (NBE) transition, namely the recombination of free excitons through an exciton–exciton collision process. On the other hand, the green band is generally explained by intrinsic defects such as oxygen vacancies and Zn interstitials. The biaxial anisotropic strain can strongly influence the valence band structure and the related selection rule [16, 17]. The

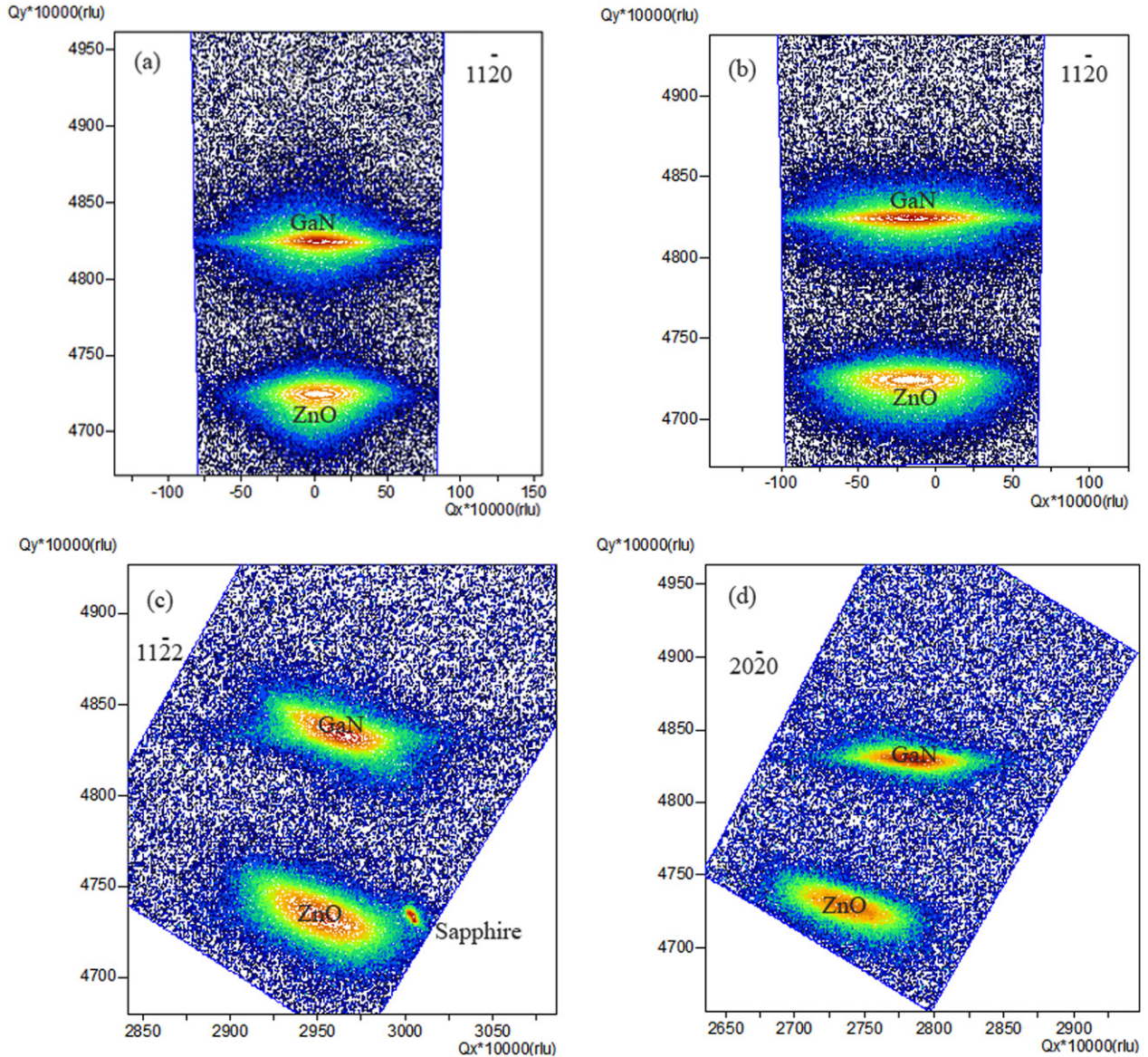


Figure 2. The combination of four RSMs of *a*-plane ZnO in reciprocal space to determine the crystallographic properties and the state of strain: (a) symmetric RSM of $(1\ 1\ \bar{2}\ 0)$ at azimuth 0° , (b) symmetric RSM of $(1\ 1\ \bar{2}\ 0)$ at azimuth 90° , (c) asymmetric RSM of $(1\ 1\ \bar{2}\ 2)$, (d) asymmetric RSM of $(2\ 0\ \bar{2}\ 0)$.

inset in figure 3 shows the high resolution PL spectra of NBE emission at 85 K. Taking into account a careful Lorentz curve fitting procedure, there are seven apparent peaks. The dominant peaks at 3.351 eV and 3.315 eV are assigned to be neutral donor-bound exciton emission (DX), free-electron-to-bound transition ($e\text{-}A_0$), respectively. The PL peak at 3.31 eV has been studied in detail by Schirra, which is ascribed to the transitions of electrons from the conduction band recombining with holes localized at the acceptor states induced by basal plane stacking faults in the ZnO films [18]. Here, the DX emission line related to donor defects presents a FWHM of approximately 21 meV, compared with the $e\text{-}A_0$ line related to acceptor defects of 38 meV. Also, there is a shoulder on the high-energy side of the DX line at 3.366 eV. Contribution from the recombination of the free exciton (FX) will progressively dominate the PL spectrum with increasing temperatures due to the thermal effect. In practice, the partial strain relaxation

induces inhomogeneities of the strain fields which broadens the excitonic line in the nonpolar orientation (FWHM ~ 17 meV). Furthermore, the emissions at 3.242 eV and 3.172 eV with a separation of 70 meV should be labelled as the first-order and second-order LO-phonon replicas of ($e\text{-}A_0$) (3.315 eV), which are relatively intense (hinting at the participation of an acceptor in this transition). This observation of associated LO-phonon replicas is typical of an electron from the conduction band with a hole bound to an acceptor state [18]. In addition, the peak at 3.122 eV is usually attributed to the native defect of zinc vacancy (V_{Zn}) [19].

To clarify the origins of these peaks, temperature-dependent PL measurements were performed in the range 85–290 K and are plotted in figure 4(a). It is remarkable that it is the $e\text{-}A_0$ and DX emissions, not the FX emission, which are clearly observed when the temperature is above 85 K, indicating the fact that the localization energy of the

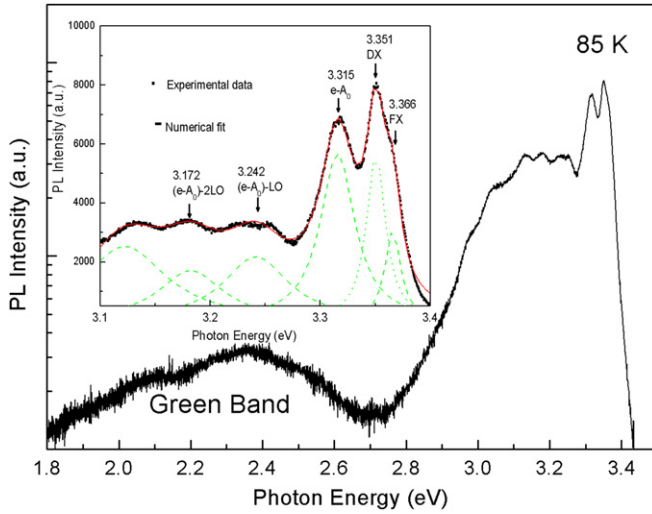


Figure 3. The typical low temperature (85 K) PL spectrum obtained from ZnO film grown on GaN/Al₂O₃ by PLD at 600 °C. The inset displays details of the NBE region by the Lorentz fitting.

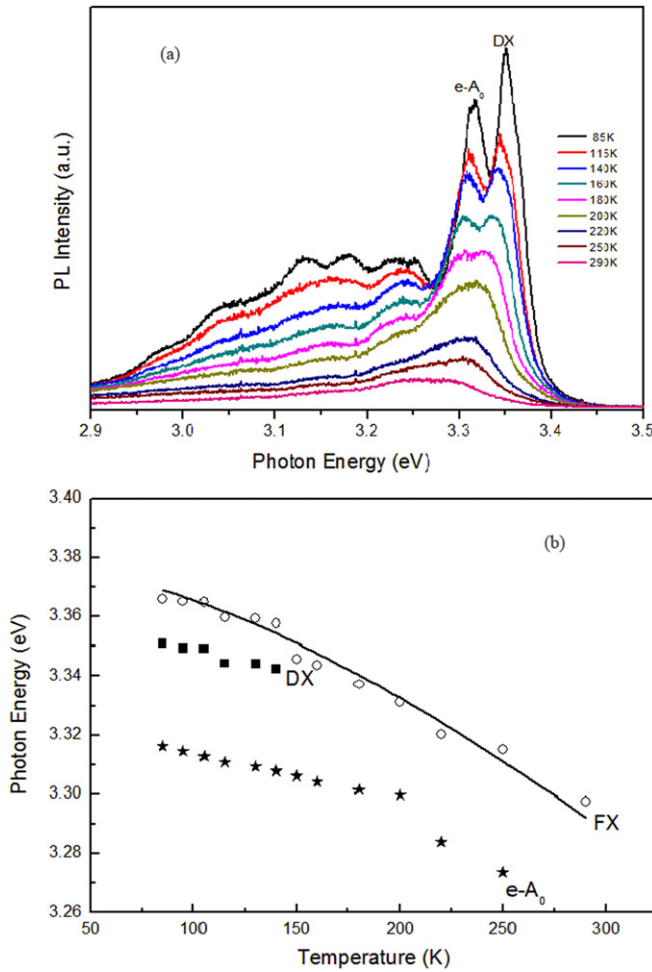


Figure 4. (a) Temperature dependence of PL spectra of the same ZnO sample ranging from 85 to 290 K. (b) Temperature dependence of the peak energy positions for FX, DX and e-A₀ emissions. The solid line curve represents the best fitting result based on Varshni's empirical model.

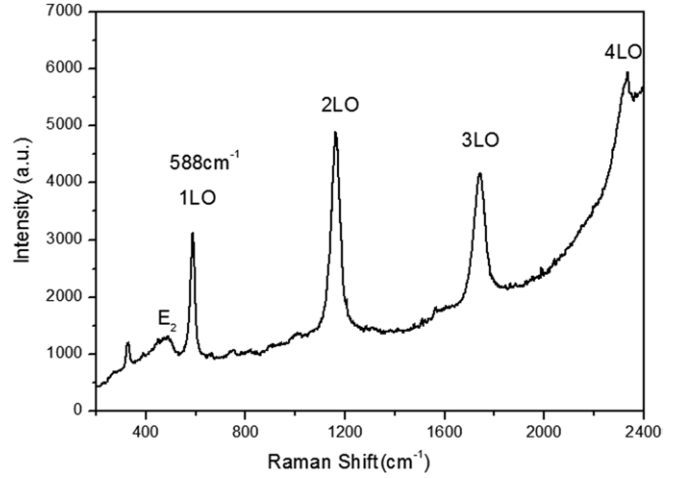


Figure 5. Resonant Raman spectrum of *a*-plane ZnO film on GaN template. *n*LO represents the *n*th longitudinal optical phonon.

DX in nonpolar ZnO is much larger than in polar ZnO. This is consistent with what has been reported in the literature for highly strained *a*-plane ZnO [17]. For $T > 150$ K, the edge emission becomes broad as the e-A₀ line merges into the low-energy tail of the FX peak. The DX emission appears to be thermally quenched above approximately 140 K, thereby the DX band is absent by superposing with the FX and preventing adequate identification of the DX position. The variation of FX peak energy position as a function of temperature is plotted, as illustrated in figure 4(b). The figure shows that the gradual red shift with increase in temperature supports its excitonic origin. Due to the temperature-induced change in lattice parameters and electron-lattice interaction, the temperature variation of the free exciton peak energy is possible to fit the following Varshni's empirical formula: $E(T) = E(0) - \alpha T^2 / (T + \beta)$, where $E(0)$, α and β are the fitting parameters [20]. The calculated temperature dependence is shown by a solid curve and fits well the experimental value in figure 4(b), where the best obtained values of $E(0)$, α and β are 3.379 eV, $8.6 \times 10^{-4} (\pm 0.5 \times 10^{-4})$ eV K⁻¹ and 542(±18) K, respectively. A slight deviation is observed due to the error in the determination of the peak position resulting from temperature broadening as explained before. These fitting values are in reasonable agreement with the literature reports for undoped bulk ZnO crystals grown by the seeded-chemical-vapour-transport method [20]. From figure 4(b), it is found that the e-A₀ peak seems to be much slower red-shifted than FX, which is a typical behaviour of free-to-bound transitions, due to the increase in the energy of the free carriers by $KT/2$ with temperature. This phenomenon has been reported in ZnO-related material systems [17, 21]. Moreover, a further study on this topic is underway in our group by time-resolved PL spectra of zinc oxide films.

The vibrational property of ZnO is further studied by the room-temperature UV RRS under 325 nm excitation; the obtained spectrum is presented in figure 5. In RRS, the excitation photon energy is resonant with the electronic interband transition energy of the wurtzite ZnO. The UV-Raman spectra were recorded with a very low laser power to avoid thermal red shift. The resonant Raman spectra of ZnO

show strong multiphonon longitudinal optical (LO) modes up to fourth order located in the high-energy side of the PL via the Frohlich mechanism, indicating a resonant enhancement effect. As seen from figure 5, the Lorentz curve fitting results show four major bands, centred at 588 cm^{-1} , 1162 cm^{-1} , 1742 cm^{-1} and 2335 cm^{-1} , respectively. They are attributed to the Raman $A_1(\text{LO})$ phonon scattering and its overtones. Indeed, centering at 588 cm^{-1} of the first-order LO phonon and the intensity ratio $I_{2\text{LO}}/I_{1\text{LO}}$ (1.5) are proved reasonable. (According to the theory of polar optical phonons in wurtzite nanocrystals, the frequency of 1LO-phonon mode in ZnO should have been 574 and 591 cm^{-1} .) Our key observation in this case is that the Raman mode frequency of the 1LO-phonon peak is different from the strain-free bulk a -plane ZnO, because of the anisotropic short-range forces in the uniaxial ZnO lattice [22]. In addition to conventional LO phonons, the peak located at about 328 cm^{-1} in figure 5 is attributed to the second-order Raman processes.

4. Conclusion

Experimental results demonstrate that high quality a -plane ($1\bar{1}20$) orientation ZnO films have been successfully grown epitaxially on a GaN template by PLD around 600°C . It is found that a nonpolar GaN template with a SiN_x nanomask is effective in improving the crystal quality of a -plane ZnO. Due to low symmetry of a -plane, the symmetric and asymmetric RSMs reveal the RC broadening behaviour and anisotropic residual strain state of the a -plane ZnO film using XRD. From the PL spectra measured at $85\text{--}290\text{ K}$, the distinct UV excitonic emission features have been observed, and their origins have been identified. Moreover, UV-Raman spectrum has shown the ZnO film with higher order LO-phonon modes up to fourth order and no TO phonons under the resonant excitation, confirming good optical quality. Further improvement of crystal quality will be possible by growing on a better crystalline GaN template and by combining with the MBE or MOCVD methods. These encouraging results and the potential for further optimization should be helpful in designing and making high efficiency nonpolar GaN-based or ZnO-based devices.

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